Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N-(3-Chlorophenyl)maleamic acid

B. Thimme Gowda,[a]* Miroslav Tokarčík,[b] K. Shakuntala,[a] Jozef Kožíšek[b] and Hartmut Fuess[c]

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, [b]Faculty of Chemical and Food Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic, and [c]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

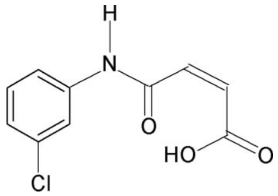
Received 1 June 2010; accepted 4 June 2010

Key indicators: single-crystal X-ray study; *T* = 295 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.028; *wR* factor = 0.081; data-to-parameter ratio = 13.4.

In the title compound, $C_{10}H_8ClNO_3$, the molecular conformation is stabilized by two intramolecular hydrogen bonds. The first is a short O—H⋯O hydrogen bond within the maleamic acid unit and the second is a C—H⋯O hydrogen bond which connects the amide group with the phenyl ring. The maleamic acid unit is essentially planar, with an r.m.s. deviation of 0.044 Å, and makes a dihedral angle of 15.2 (1)° with the phenyl ring. In the crystal, intermolecular N—H⋯O hydrogen bonds link the molecules into *C*(7) chains running [010].

Related literature

For studies on the effect of ring- and side-chain substitutions on the crystal structures of amides, see: Gowda *et al.* (2010*a*,*b*); Prasad *et al.* (2002); Shakuntala *et al.* (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{10}H_8ClNO_3$
M_r = 225.62
Monoclinic, $P2_1/c$
a = 10.7779 (3) Å
b = 13.2103 (4) Å
c = 7.1372 (2) Å
β = 104.976 (3)°
V = 981.69 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 295 K
0.55 × 0.09 × 0.06 mm

Data collection

Oxford Diffraction Gemini R, CCD diffractometer
Absorption correction: analytical (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.852, T_{max} = 0.982
15632 measured reflections
1829 independent reflections
1533 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.081
S = 1.08
1829 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O2—H2*A*⋯O1	0.90	1.60	2.4992 (14)	176
N1—H1*N*⋯O3^{i}	0.86	1.99	2.8403 (15)	172
C6—H6⋯O1	0.93	2.31	2.8658 (16)	118

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2002); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2009) and *WinGX* (Farrugia, 1999).

MT and JK thank the Grant Agency of the Slovak Republic (VEGA 1/0817/08) and the Structural Funds, Interreg IIIA, for financial support in purchasing the diffractometer. KS thanks the University Grants Commission, Government of India, New Delhi, for the award of a research fellowship under its faculty improvement program.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2280).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Brandenburg, K. (2002). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gowda, B. T., Tokarčík, M., Kožíšek, J., Shakuntala, K. & Fuess, H. (2010*a*). *Acta Cryst.* E**66**, o51.
Gowda, B. T., Tokarčík, M., Shakuntala, K., Kožíšek, J. & Fuess, H. (2010*b*). *Acta Cryst.* E**66**, o1554.
Oxford Diffraction (2009). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Prasad, S. M., Sinha, R. B. P., Mandal, D. K. & Rani, A. (2002). *Acta Cryst.* E**58**, o1296–o1297.
Shakuntala, K., Gowda, B. T., Tokarčík, M. & Kožíšek, J. (2009). *Acta Cryst.* E**65**, o3119.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2010). E**66**, o1643 [**doi:10.1107/S1600536810021446**]

N-(3-Chlorophenyl)maleamic acid

B. T. Gowda, M. Tokarcík, K. Shakuntala, J. Kozísek and **H. Fuess**

Comment

As a part of studying the effect of ring and side chain substitutions on the crystal structures of biologically significant amides (Gowda *et al.*, 2010*a,b*; Prasad *et al.*, 2002; Shakuntala *et al.*, 2009), the crystal structure of *N*-(3-chlorophenyl)maleamic acid (I) has been determined (Fig. 1).The conformations of the N—H and the C=O bonds in the amide segment are *anti* to each other. The conformation of the N—H bond is also *anti* to the *meta*-Cl group in the phenyl ring.In the maleamic acid moiety, the amide C=O bond is *anti* to the adjacent C—H bond, while the carboxyl C=O bond is *syn* to the adjacent C—H bond. The observed rare *anti* conformation of the C=O and O—H bonds of the acid group is similar to that observed in *N*-(2-methylphenyl)-maleamic acid (Gowda *et al.*, 2010*b*), *N*-(2,5-dichlorophenyl)-maleamic acid (Shakuntala *et al.*, 2009) and *N*-(3,5-dichlorophenyl)- maleamic acid (Gowda *et al.*, 2010*a*).

The molecular structure of (I) is stabilized by two intramolecular hydrogen bonds (Figure 1): the first is a short O—H···O hydrogen bond within maleamic acid unit and the second is a C—H···O hydrogen bond which connects the amide group with the phenyl ring. Amidic O1 atom acts as bifurcated acceptor of O2—H2A···O1 and C6—H6···O1 intramolecular hydrogen bonds (Table 1).The maleamic acid unit is essentially planar, with r.m.s.deviation of 0.044 Å and makes dihedral angle of 15.2 (1)° with the phenyl ring. The torsion angle C1—N1—C5—C6 = -17.6 (2)° defines the orientation of the phenyl ring towards the central amide group –NHCO. The molecular structure is stabilized by two types intramolecular C—H···O and O—H···O interactions with H···O distances of 1.60 and 2.31 Å respectively and one intermolecular N—H···O hydrogen bonds link the molecules into chains with graph-set notation C(7) (Bernstein *et al.*, 1995) running along the [0 1 0] direction, Table 1, Figure 2.

Experimental

The solution of maleic anhydride (0.025 mol) in toluene (25 ml) was treated dropwise with the solution of 3-chloroaniline (0.025 mol) also in toluene (20 ml) with constant stirring. The resulting mixture was warmed with stirring for over 30 min and set aside for an additional 30 min at room temperature for completion of the reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 3-chloroaniline. The resultant solid *N*-(3-chlorophenyl)maleamic acid was filtered under suction and washed thoroughly with water to remove the unreacted maleic anhydride and maleic acid. It was recrystallized to constant melting point from ethanol. The purity of the compound was checked by elemental analysis and characterized by its infrared spectra.

Rod like colourless single crystals used in X-ray diffraction studies were grown in an ethanol solution by slow evaporation at room temperature.

Refinement

All H atoms were found in difference maps and further treated as riding atoms with C—H = 0.93 Å, N—H = 0.86Å and O—H = 0.90 Å. The U_{iso}(H) values were set at $1.2U_{eq}$(C aromatic, N) and $1.5U_{eq}$(O).

Figures

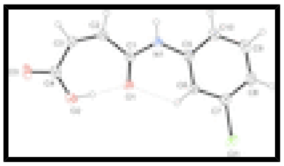


Fig. 1. Molecular structure of (I) showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii and a short intramolecular O—H···O bond as dashed line.

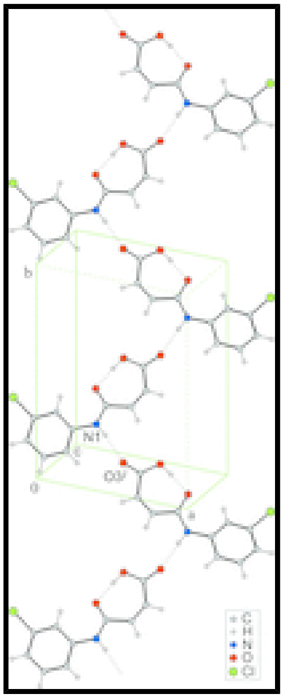


Fig. 2. Part of crystal structure of (I) showing one-dimensional chain of molecules extending along the [0 1 0] direction and linked by intermolecular N–H···O hydrogen bonds. Hydrogen bonds are shown as dashed lines. Symmetry code (i): -*x* + 1, *y* - 1/2, -*z* + 3/2.

N-(3-Chlorophenyl)maleamic acid

Crystal data

$C_{10}H_8ClNO_3$

$M_r = 225.62$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.7779$ (3) Å

$b = 13.2103$ (4) Å

$c = 7.1372$ (2) Å

β = 104.976 (3)°

$V = 981.69$ (5) Å^3

$Z = 4$

$F(000) = 464$

$D_x = 1.527$ Mg m^{-3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 8764 reflections

θ = 2.0–29.5°

μ = 0.37 mm^{-1}

$T = 295$ K

Rod, colourless

0.55 × 0.09 × 0.06 mm

Data collection

Oxford Diffraction Gemini R, CCD diffractometer

graphite

1829 independent reflections

1533 reflections with $I > 2\sigma(I)$

Detector resolution: 10.434 pixels mm^{-1}

ω scans

Absorption correction: analytical (*CrysAlis PRO*; Oxford Diffraction, 2009)

$T_{\min} = 0.852$, $T_{\max} = 0.982$

15632 measured reflections

$R_{\text{int}} = 0.027$

$\theta_{\max} = 25.5°$, $\theta_{\min} = 2.0°$

$h = -13 \rightarrow 13$

$k = -16 \rightarrow 16$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.081$

$S = 1.08$

1829 reflections

136 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.0776P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.16$ e Å^{-3}

$\Delta\rho_{\min} = -0.16$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.31064 (12)	0.29347 (9)	0.57205 (19)	0.0320 (3)
C2	0.44462 (13)	0.28772 (10)	0.6940 (2)	0.0372 (3)
H2	0.4774	0.2228	0.7228	0.045*
C3	0.52416 (13)	0.36307 (11)	0.7676 (2)	0.0401 (3)
H3	0.6043	0.3415	0.8403	0.048*
C4	0.50988 (14)	0.47472 (11)	0.7564 (2)	0.0426 (4)
C5	0.12674 (13)	0.18104 (10)	0.42427 (19)	0.0319 (3)
C6	0.03012 (12)	0.25287 (10)	0.38134 (18)	0.0328 (3)
H6	0.0463	0.3197	0.4214	0.039*
C7	−0.09097 (13)	0.22302 (11)	0.27750 (19)	0.0367 (3)
C8	−0.11862 (14)	0.12464 (12)	0.2179 (2)	0.0444 (4)
H8	−0.201	0.1061	0.149	0.053*

C9	−0.02125 (16)	0.05452 (12)	0.2627 (2)	0.0509 (4)
H9	−0.0383	−0.0123	0.2236	0.061*
C10	0.10121 (14)	0.08138 (11)	0.3646 (2)	0.0432 (4)
H10	0.1662	0.0331	0.3932	0.052*
N1	0.25350 (10)	0.20331 (8)	0.53348 (16)	0.0346 (3)
H1N	0.2998	0.1519	0.5813	0.042*
O1	0.25666 (9)	0.37411 (7)	0.51038 (16)	0.0479 (3)
O2	0.40462 (10)	0.51612 (8)	0.65145 (18)	0.0565 (3)
H2A	0.3485	0.4673	0.5973	0.085*
O3	0.59801 (11)	0.52618 (9)	0.84566 (19)	0.0666 (4)
Cl1	−0.21185 (3)	0.31393 (3)	0.22262 (6)	0.05280 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0286 (7)	0.0273 (7)	0.0383 (7)	0.0013 (5)	0.0054 (6)	0.0006 (6)
C2	0.0316 (7)	0.0292 (7)	0.0465 (8)	0.0040 (6)	0.0023 (6)	0.0016 (6)
C3	0.0284 (7)	0.0370 (8)	0.0484 (8)	0.0011 (6)	−0.0016 (6)	0.0001 (6)
C4	0.0387 (8)	0.0345 (8)	0.0524 (9)	−0.0072 (6)	0.0080 (7)	−0.0053 (6)
C5	0.0306 (7)	0.0300 (7)	0.0336 (7)	−0.0038 (5)	0.0054 (6)	0.0015 (5)
C6	0.0314 (7)	0.0286 (7)	0.0358 (7)	−0.0026 (6)	0.0042 (6)	0.0003 (5)
C7	0.0307 (7)	0.0427 (8)	0.0344 (7)	−0.0021 (6)	0.0045 (6)	0.0031 (6)
C8	0.0362 (8)	0.0462 (9)	0.0461 (8)	−0.0128 (7)	0.0019 (6)	−0.0033 (7)
C9	0.0523 (10)	0.0333 (8)	0.0626 (10)	−0.0119 (7)	0.0069 (8)	−0.0096 (7)
C10	0.0411 (8)	0.0294 (7)	0.0560 (9)	−0.0008 (6)	0.0070 (7)	−0.0021 (6)
N1	0.0293 (6)	0.0257 (6)	0.0444 (7)	0.0022 (5)	0.0014 (5)	0.0023 (5)
O1	0.0340 (5)	0.0281 (5)	0.0707 (7)	0.0001 (4)	−0.0058 (5)	0.0070 (5)
O2	0.0445 (6)	0.0282 (6)	0.0871 (9)	−0.0008 (5)	−0.0004 (6)	−0.0016 (5)
O3	0.0538 (7)	0.0450 (7)	0.0886 (9)	−0.0191 (6)	−0.0038 (6)	−0.0113 (6)
Cl1	0.0326 (2)	0.0540 (3)	0.0625 (3)	0.00544 (16)	−0.00438 (17)	0.00293 (18)

Geometric parameters (Å, °)

C1—O1	1.2389 (16)	C6—C7	1.3813 (18)
C1—N1	1.3364 (17)	C6—H6	0.93
C1—C2	1.4828 (19)	C7—C8	1.376 (2)
C2—C3	1.330 (2)	C7—Cl1	1.7404 (15)
C2—H2	0.93	C8—C9	1.374 (2)
C3—C4	1.483 (2)	C8—H8	0.93
C3—H3	0.93	C9—C10	1.379 (2)
C4—O3	1.2073 (18)	C9—H9	0.93
C4—O2	1.3069 (18)	C10—H10	0.93
C5—C6	1.3834 (19)	N1—H1N	0.86
C5—C10	1.3891 (19)	O2—H2A	0.90
C5—N1	1.4178 (17)		
O1—C1—N1	122.96 (12)	C5—C6—H6	120.8
O1—C1—C2	123.32 (12)	C8—C7—C6	122.32 (13)
N1—C1—C2	113.72 (11)	C8—C7—Cl1	119.50 (11)

C3—C2—C1	128.61 (13)	C6—C7—Cl1	118.19 (11)
C3—C2—H2	115.7	C9—C8—C7	118.25 (13)
C1—C2—H2	115.7	C9—C8—H8	120.9
C2—C3—C4	132.50 (13)	C7—C8—H8	120.9
C2—C3—H3	113.7	C8—C9—C10	121.25 (14)
C4—C3—H3	113.7	C8—C9—H9	119.4
O3—C4—O2	120.99 (14)	C10—C9—H9	119.4
O3—C4—C3	118.36 (14)	C9—C10—C5	119.50 (14)
O2—C4—C3	120.65 (13)	C9—C10—H10	120.2
C6—C5—C10	120.27 (12)	C5—C10—H10	120.2
C6—C5—N1	122.87 (11)	C1—N1—C5	128.71 (11)
C10—C5—N1	116.85 (12)	C1—N1—H1N	115.6
C7—C6—C5	118.41 (12)	C5—N1—H1N	115.6
C7—C6—H6	120.8	C4—O2—H2A	109.5

O1—C1—C2—C3	5.1 (2)	Cl1—C7—C8—C9	179.82 (12)
N1—C1—C2—C3	−175.30 (14)	C7—C8—C9—C10	0.0 (2)
C1—C2—C3—C4	0.0 (3)	C8—C9—C10—C5	0.4 (2)
C2—C3—C4—O3	177.02 (16)	C6—C5—C10—C9	−0.2 (2)
C2—C3—C4—O2	−3.3 (3)	N1—C5—C10—C9	178.16 (13)
C10—C5—C6—C7	−0.4 (2)	O1—C1—N1—C5	−1.2 (2)
N1—C5—C6—C7	−178.62 (12)	C2—C1—N1—C5	179.26 (12)
C5—C6—C7—C8	0.8 (2)	C6—C5—N1—C1	−17.6 (2)
C5—C6—C7—Cl1	−179.60 (10)	C10—C5—N1—C1	164.11 (13)
C6—C7—C8—C9	−0.5 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···O1	0.90	1.60	2.4992 (14)	176
N1—H1N···O3^{i}	0.86	1.99	2.8403 (15)	172
C6—H6···O1	0.93	2.31	2.8658 (16)	118

Symmetry codes: (i) $-x+1$, $y-1/2$, $-z+3/2$.

Fig. 1

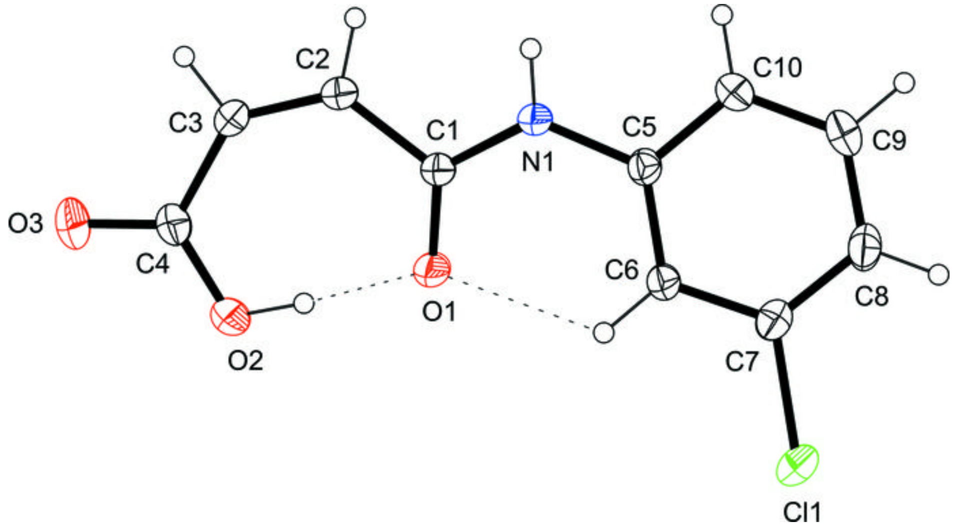


Fig. 2